ANALYTICAL CHEMISTRY DATA MANAGEMENT AND REVIEW FOR SOIL, FOODSTUFFS AND BIOTA

Purpose

This Meteorology and Air Quality Group (MAQ) procedure describes the process for receiving, uploading, and archiving analytical chemistry data; evaluating analytical chemistry quality; checking the resulting chemistry data packages for completeness and usability; and conducting validation/verification of both electronic and hardcopy data from current sources.

Scope

This procedure applies to the analytical chemistry coordinator assigned to evaluate Soil, Foodstuffs, and Biota analytical data.

In this procedure

Hazard Control Plan

The hazard evaluation associated with this work is documented in MAQ-Gen-Office Work.

Signatures

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04/01/04

General information about this procedure

Attachments

This procedure has the following attachments:

Number	Attachment Title	No. of pages
1	Checklist for Completeness of Data Package	1
2	Soil, Foodstuffs, and Biota Analytical Data Validation and Verification Database Inspect	1
3	QC Evaluations Performed	1

History of revision

This table lists the revision history and effective dates of this procedure.

Revision	Date	Description of Changes
0	3/29/04	New document.

Who requires training to this procedure?

The following MAQ personnel require training before implementing this procedure:

- Analytical chemistry data reviewers
- Analytical Chemistry Coordinator

Training method

The initial training method for this procedure is **on-the-job** training by a previously trained individual, and is documented in accordance with the procedure for training (MAQ-024).

Annual retraining is required and will be by self-study (“reading”) training.

General information, continued

Prerequisites

In addition to training to this procedure, the following training is also recommended prior to performing this procedure:

- Education and/or experience in compliance/surveillance-oriented analytical chemistry
- Familiarity with Microsoft Access
- Familiarity with the operation of the Soil, Foodstuffs, and Biota database

Definitions specific to this procedure

Statement of Work (SOW): A list of specifications and requirements which analytical laboratories must meet in order to do work for MAQ (prepared according to MAQ-036).

Data Package: A hardcopy report from an analytical laboratory on a single set of chemical analyses, which contains the material specified in the SOW and sufficient documentation to allow an appropriate professional, at a substantially different time and location, to ascertain:

- what analyses were performed and what results were obtained
- that the data had acceptable properties (such as accuracy, precision, MDA)
- where, when, and by whom the analyses were performed
- that the analyses were done under acceptable conditions (such as calibration, control, custody, using approved procedures, and following generally approved good practices)
- that the MAQ SOW was otherwise followed.

Defensible Data Package: A data package which the MAQ analytical chemistry coordinator and the QA Officer believe sufficient (based on EPA Contract Laboratory Program and best professional judgment) to prove the validity of chemistry results.

Usability: A qualitative decision process whereby the decision-makers evaluate the achievement of data quality objectives and determine whether the data may be used for the intended purpose. Three levels or classes of data quality are used:

- Accepted: Data conform to all requirements, all quality control criteria are met, methods were followed, and documentation is complete.
- Qualified: Data conform to most, but not all, requirements, critical QC criteria are met, methods were followed or had only minor deviations, and critical documentation is complete.

General information, continued

Definitions, continued

- Rejected: Data do not conform to some or all requirements, critical QC criteria are not met, methods were not followed or had significant deviations, and critical documentation is missing or incomplete.

Electronic Data Deliverable (EDD): The computer-compatible file that is delivered to RRES-MAQ from the analytical laboratory, in the SOW-specified format, via Internet, e-mail, or diskette from which analytical chemistry data may be uploaded directly into the databases.

Validation: A systematic process for reviewing a body of data or a report against a set of criteria to provide assurance that the data or report are adequate for their intended use. Validation consists of data reviewing, screening, checking, auditing, verification, certification, and review.

Verification: The act of reviewing, inspecting, testing, checking, auditing, or otherwise determining and documenting whether items, processes, services, or documents conform to specified requirements.

References

The following documents are referenced in this procedure:

- MAQ-024, "Personnel Training"
- MAQ-026, "Deficiency Reporting and Correcting"
- MAQ-036, "Preparing Statements of Work for Analytical Chemistry"
- MAQ-SFB, "Quality Assurance Project Plan for the Soil, Foodstuffs, and Biota Monitoring Project"

Note

Actions specified within this procedure, unless preceded with "should" or "may," are to be considered mandatory guidance (i.e., "shall").

Background

Description of data upload and verification

Requirements for chemical analyses are described in the data quality objectives (DQO) section of the Quality Assurance Project Plan for the Soil, Foodstuffs, and Biota Monitoring Project (MAQ-SFB). Data quality objectives are translated into procurement needs and related Statements of Work (SOW) according to MAQ-033. Data received from all internal and external chemistry laboratories under these SOWs are uploaded electronically and inspected to determine if they meet MAQ specifications. This inspection includes checking the data package against checklists to ensure that:

- the data package contains the components specified in statements of work,
- all of the requested analyses were performed for all samples,
- the data are of a quality adequate for the use which MAQ intended.

All manually entered data (100%) and a portion of the electronic data (minimum of 10%) are verified against the hard copy to ensure exact reproduction of the analytical concentrations, and the data usability are evaluated for acceptance, qualification, or rejection. The analytical data are evaluated to ensure usability and electronic forms are verified against hardcopy data packages, and then archived to protect their integrity.

Description of technical review validation

With the exception of soils (the soils are reported on a dry weight basis), all foodstuffs and biota are analyzed and reported on an ash weight basis and are converted to a dry weight basis for reporting purposes. Data will be compared to

- determine whether an on-site or perimeter value is detectable (when the result is higher than the minimum detectable concentration and greater than three times the analytical uncertainty) or nondetectable, and
- determine whether a detectable result is greater than the regional statistical reference level (RSRL) (the RSRL is based on the average and standard deviation of the last five years of regional data).

The technical reviewer determines whether a detectable soil result is greater than the screening action level (SAL) and the results are posted to the Access databases. Ultimately, all electronic data are archived into limited-access tables to ensure their integrity. All stages of the process are tracked electronically within the Soil, Foodstuffs, and Biota database.

Processing and evaluating the EDD for Soil, Foodstuffs, and Biota analytical chemistry data

Upload EDD

EDDs may be received from both internal and external analytical chemistry laboratories. Format and content requirements are specified in each individual Statement of Work prepared according to MAQ-033. Each EDD requires specific software to enable it to be incorporated into the existing databases. Upload these EDDs according to these detailed processes as soon after receipt as practical.

Prepare checklist for deliverables

When new services are procured, prepare a checklist as needed to evaluate the completeness of any deliverables. See the chapter "Preparing checklists for deliverables" in MAQ-033.

Evaluate against SOW requirements

After uploading data received electronically, evaluate these deliverables using software to ensure that the major components are the same as those usually received or required by the SOW.

Perform QC evaluation of EDD

The **database administrator** runs appropriate automated queries on the uploaded data to asses the data against the criteria listed in Attachment 1, and forwards the output to the project leader for technical evaluation.

The **project leader** documents the evaluation by signing the query or report printouts.

Resolution

When expected components are missing or errors are detected, the **database administrator** or the **project leader** contacts the lab immediately and requests that a revised EDD be sent expeditiously. Also document the problem by preparing a deficiency report according to MAQ-026.

Evaluating completeness

Evaluate data package completeness against completeness checklist

After receiving the final hard-copy data package and while the technical reviewer is reviewing the preliminary concentrations and QC evaluations, the **analytical chemistry coordinator** uses the appropriate completeness checklist (prepared as described in the chapter *Preparing checklists for deliverables* in MAQ-033; see Attachment 1 for an example) to evaluate the deliverable. If the data are of a frequently purchased type, review to ensure that the major package components are the same as those usually received.

Resolution

When expected components are missing, contact the lab immediately and request that the missing components be sent expeditiously. Also document the problem by preparing a deficiency report according to MAQ-026.

Custody errors

Custody errors are those which make it difficult to demonstrate that the samples that were shipped by MAQ were the same as those analyzed by the lab. Examples include:

- MAQ or lab staff not signing and dating chain of custody forms
- Loss or miscounting by MAQ or the lab
- Misidentifying by MAQ or the lab
- Lost samples
- Delivery to the wrong site or person

Document all custody errors with an MAQ Deficiency Report (MAQ-026). Resolution will require coordination with the lab. If new analyses are necessary, ship the new samples under a new chain of custody.

Purpose of Soil, Foodstuffs, and Biota analytical chemistry data evaluation

The data evaluation process determines whether chemical analyses data meet the data quality objectives specified in the quality plan (ECO-SFB). All data will be evaluated for one of three outcomes: *accept*, *qualify*, or *reject*. For qualified and rejected data, an explanation must be included in the database.

Evaluating completeness, continued

Steps to evaluate data

Follow the steps below to evaluate the Soil, Foodstuffs, and Biota data:

Step	Action
1	Using the appropriate sample checklist(s) prepared according to the chapter *Preparing checklists for deliverables* in MAQ-033 (see Attachment 2 for an example), evaluate for completeness. Each analytical data element should have a value. • For all missing data, ensure an explanation is recorded in the database and label the record as "rejected." If a missing datum can be located, enter the correct value, label the datum "qualified" in the database, and enter the reason for qualification. • If data errors are identified, contact the lab and negotiate for a corrected report. Label data as "rejected" pending resolution with the laboratory.
2	Using the appropriate sample checklist(s) and output from custom database queries, look for values within the expected range. For example, the expected range might be a nominal value with a range of possible values or an MDA which represents a particular dose cutoff (e.g., 0.1 mrem). Use historical ranges for concentrations at each station (calculated by a custom database query) to identify potentially suspect data points for further inspection and validation. The MDA should also be evaluated against the requirements in the SOW to ensure contractual compliance.
3	As a result of step 2, if the element is outside its range of normal values or significantly above the required MDA, identify the record as "qualified." Perform further validation and verification. Consult with the vendor to determine what conditions at their laboratory may have resulted in the data value reported. Examine field records to identify possibilities of contamination during handling. Label any amended analytical records as "qualified" (enter a "Q" in the analytical data qualification field) and describe in the table's comment field the amendments made. Prepare and reference a separate memo if necessary to provide sufficient detail.
4	If a "qualified" data point cannot be logically amended or explained, it may be labeled as "rejected" (enter a "R" in the analytical data qualification field) and the reasons for rejection must be provided in the table's comment field. Prepare and reference a separate memo if necessary to provide sufficient detail.

Evaluating completeness, continued

Develop custom queries for data evaluation	Upon request of the project leader or technical reviewer for FSB data, develop custom queries to automate the checking and calculation of the data against technical evaluation criteria.
Run custom queries for data evaluation	Run any custom queries that perform technical evaluations of the data and forward these data to the technical reviewer for technical evaluation.

Technical review of data

Technical review

The **technical reviewer** responsible for routine review of these data conducts a review for the following:

- Data are properly converted to a dry weight basis or are otherwise properly converted to needed units.
- Determine whether an on-site or perimeter value is detectable (when the result is higher than the minimum detectable concentration and greater than three times the analytical uncertainty).
- Determine whether a detectable result is greater than the regional statistical reference level (RSRL) (the RSRL is based on the average and standard deviation of the last five years of regional data).
- For soils, determine whether a detectable result is greater than the screening action level (SAL).
- Determine which data points may need to be qualified or rejected based on field collection data or other information.

Document needed changes and forward for implementation

If changes to the database are needed, the **reviewer** documents the needed changes in a memo or form and forwards the form to the database coordinator or analytical chemistry coordinator for implementation in the database.

Making technical reviewer changes to the database

Technical reviewer action implementation, final data archiving and public release

After receiving the results of the technical review, the **database coordinator** or **analytical chemistry coordinator** implements the indicated changes in acceptance outcomes to both field and analytical data. The data are archived in limited access tables for protection from inadvertent modification.

Steps to implement technical review input

Perform the following steps to implement the recommendations and changes from the technical reviewer:

Step	Action
1	After the technical reviewer returns a formal memo listing the changes to be made, implement the recommended actions in the database and document the reasons in the comment field.
2	When both the validation and verification and technical review process are complete, archive both field and analytical chemistry data. These become the official data for use in published surveillance reports and for release to the public.
3	Publish fully approved data to the MAQ WWW homepage.

Records resulting from this procedure

Records

The following records generated as a result of this procedure are to be submitted **within 3 weeks of their receipt or generation** as records to the records coordinator:

- Soil, Foodstuffs, and Biota Completeness of Data Package form; completed, signed, and dated
- Soil, Foodstuffs, and Biota Field Data Validation and Verification Database inspection form; completed, signed and dated.
- Soil, Foodstuffs, and Biota Analytical Data Validation and Verification Database Inspection form; completed, signed, and dated.
- Copy of final laboratory data package
- Deficiency reports resulting from chain-of-custody problems, if any
- MAQ internal memos documenting data quality evaluation, data validation, and initial concentration calculations

The following electronic records generated as a result of this procedure are to be contained within their respective Microsoft Access databases:

- entries in Soil, Foodstuffs, and Biota database for all accepted, qualified, and rejected data from both field and analytical processes.

QC EVALUATIONS PERFORMED

Type of Data	Evaluation Performed	Acceptance Criteria
All	LCS recovery check	100 ±10%
All	Process Blank (PB)	See Control Criteria below
All	Matrix Blank (MB)	See Control Criteria below
All	Trip Blank (TB)	See Control Criteria below
Alpha, Beta, H-3, alpha isotopics and Be	Matrix Replicate evaluation	For analytically significant, positive results, similar to control criteria below.
Gamma	Matrix Replicate evaluation	Qualitative agreement (within a factor of 5) for analytically insignificant results (i.e. less-than values).
Be, H-3 and alpha isotopics	Matrix Spike	100 ±10% of added spike
All	MDA achieved	All samples below SOW specification
All	Missing Field or Analytical data	No missing data for actual field samples
Tritium	Collection efficiency	Between 50 and 130 % of theoretical
Gamma	"Naturals"	All should have positive results
Gamma	"Artificials"	Compare calculated dose to 0.5 mrem target
All	Analytical Completeness	80% successful analysis of valid samples
Alpha/beta, H-3, alpha isotopics	Action Level Comparison	< 100% of target value

General Control criteria:
"Under control" is within <= 2s of annual mean for that QC type
"Warning" is between 2s and 3s of annual mean for that QC type
"Out of control" is >= 3s of annual mean for that QC type

Meteorology and Air Quality Group

Soil, Foodstuffs, and Biota Analytical Data Validation and Verification Database Inspect

This form is from MAQ-712

Gross Alpha/Beta (8/1/99 version)

Soil, Foodstuffs, and Biota Sample Group #: _____

Data Element Inspected (Database location)	Complete and Correct in referenced Access table	Comments
Data Package Completeness check performed	Y - N	
Data V&V method used: All manually entered 10% of EDD	 Y - - NA Y - - NA	
ANAL DATA FOR V&V table		
Soil, Foodstuffs, and Biota Sample Period #	Y - N - NA	
Soil, Foodstuffs, and Biota Sample ID number	Y - N - NA	
Location # = Soil, Foodstuffs, and Biota ID after decimal point?	Y - N - NA	
Anal Lab sample ID	Y - N - NA	
Analysis	Y - N - NA	
< in Symbol field, if approp.	Y - N - NA	
Result	Y - N - NA	
Uncertainty	Y - N - NA	
Units	Y - N - NA	
MDA	Y - N - NA	
Comment	Y - N - NA	
Master Site Numbers	Y - N	
Data Qualifiers in use	Y - N	
SAMPLE & DATA TRACKING table		
Anal. Lab code entered	Y - N	
Date Submitted	Y - N - NA	
Date Received	Y - N - NA	
Anal Lab SDG #	Y - N - NA	
Lab Analytical Procedure #	Y - N	
Filter fractions complete	Y - N	
Uncer & MDA precision chara.	Y - N - NA	

Verified by: _____ Date: _____

ATTACHMENT 3

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Checklist for Completeness of Data Package

This form is from MAQ-712

Soil, Foodstuffs, and Biota Sample Group #:_____

Inspection Criterion	Criter met?	Comments
Was analytical lab required to work to the above-listed standard by contract?	Y N NA	
Was an acceptable EDD received within 14 days of lab receipt of samples	Y N NA	
Final Data package received within 30 days of sample arrival at analytical lab?	Y N NA	Date sub: Date rcd:
Each page of each data package sequentially numbered.	Y N NA	
Narrative comments on the analysis of each sample group in cover letter or memo?	Y N NA	
Positive sample id in all tables and reports.	Y N NA	
Positive indication of signatures/initials at each work and review stage.	Y N NA	
Data received for each sample on C-of-C.	Y N NA	
Summary of sample results (to include customer id, sample delivery group or request number, lab id, isotope/analysis, analyte concentration, analyte uncertainty and MDA in the same appropriate units, counting times, and dates of analysis); an individual summary provided for each sample.	Y N NA	
Individual summary of each QA/QC sample (same parameters as sample results); QA/QC samples will include, at a minimum of one each of the following for every 20 field samples: a Laboratory Control Sample (LCS), a detector blank, a matrix blank and a matrix spike.	Y N NA	
Known values for all QA/QC samples?	Y N NA	
Individual sample raw data and individual spectral plots showing regions of interest (ROI) integrated for each gamma isotope.	Y N NA	
Individual QA/QC raw data and individual spectral plots showing ROI integrated for each isotope.	Y N NA	
Individual detector efficiencies and backgrounds.	Y N NA	
Laboratory bench sheets with sample of any manual calculations done.	Y N NA	
Evidence of NIST-traceable calibration standards.	Y N NA	
Copies of the most recent applicable MDA study results, initial calibration and recalibration.	Y N NA	
Chain of custody form.	Y N NA	
All equations used to calculate MDAs or sample results either in datapackage or published analytical procedures.	Y N NA	
Actual concentrations include negative values, rather than some form of "not detected" (less-thans are permitted).	Y N NA	
Uncertainties (identified appropriately as 1, 2, or 3 sigma in the final data package).	Y N NA	

Verified by: _____ Date: _____